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New Hyperthermal Thermosetting Heterocyclic Polymers

Polyimidazopyrrolone ("pyrrone") polymers, formed by the condensation reaction of aromatic dianhydrides with aromatic tetraamines in various solvents, are excellent thermosetting resins that form moldings resisting degradation in air and retaining great strength at temperatures between 400° and 700°F. Varnishes made from the resins can be used for coating fabrics or fibers in the manufacture of reinforced moldings or laminates. The resins have good insulating properties, are easy to mold, and make good protective coatings.

One such resin was prepared by addition of 3,3',4,4'-benzophenonetetracarboxylic dianhydride (96.6 g, 0.3 mole) to 500 ml of hot absolute ethanol. Upon the drop-by-drop addition of 20 ml of concentrated sulfuric acid, the dianhydride dissolved. After the reaction mixture was stirred at reflux for 20 hours, ethanol was distilled until the temperature of the mixture rose above 100°C. Dissolved in ether, the residue was washed several times with water; it was then shaken with a 10% solution of sodium bicarbonate until acidification of the bicarbonate washes failed to produce a precipitate. After drying over anhydrous magnesium sulfate, the ether was evaporated and a product weighing 89.3 g (0.19 mole) was collected—63% of the theoretical product.

This product was dissolved in 500 ml of absolute ethanol, and 3,3'-diaminobenzidine (40.5 g, 0.19 mole) was added to the refluxing solution. Ethanol was distilled from the resultant slurry until the temperature rose to 150°C at which point the mixture became a viscous melt. While being stirred the mixture was

heated further to about 200°C; after 90 min it became too viscous for stirring. When cooled the melt became a brittle glass, weighing 108.4 g, that could be ground and used as a molding powder. When dissolved in *N,N*-dimethylacetamide, the partially reacted product served as a varnish. Fiberglass laminates had flexural strengths exceeding 70,000 lb/in², and moduli exceeding 3×10^6 lb/in² were readily achieved.

Notes:

1. The chemical and aircraft industries may be interested.
2. The following documentation may be obtained from:

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Reference: NASA-CR-1310 (N69-26448),
Development and Processing of Pyrrone
Polymers

Patent status:

Inquiries about obtaining rights for the commercial use of this invention may be made to NASA, Code GP, Washington, D.C. 20546.

Source: N. Bilow, A.L. Landis
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